X-ray tomography :
a tool for revealing local distribution of liquid in structured packed columns

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PEPs CHEMICAL ENGINEERING

LIÈGE université

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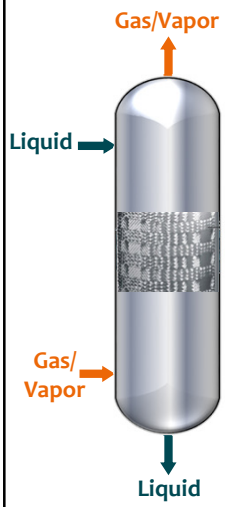
Structured packed columns



Packed column applications

- Gas-liquid absorption ⇔ CO_2 capture
- Solvent stripping ⇔ regeneration
- Distillation

High performance structured packings

- High void fraction
  ➔ low pressure drop ⇔ high capacity
- High specific surface area
  ➔ high mass transfer properties

Structured packed columns

Liquid flow hydrodynamics

- Liquid holdup
- Gas – liquid interfacial surface area
- Liquid flow morphology
  (films, rivulets…)

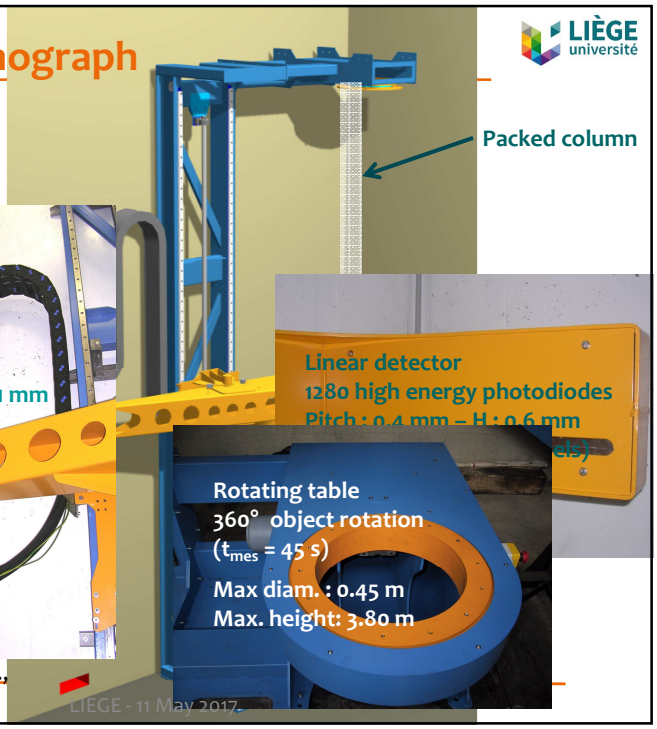
Reliable predictive models

⇔ **Characterisation** of the **fluid distribution** down to a very **local scale**

➔ **X-ray tomography**

ULG X-ray tomograph

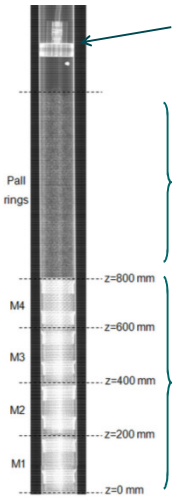
Operating modes
1- tomographic mode
2- radiographic mode



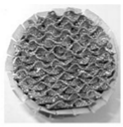
Toye et al., Meas. Sci. Technol., 2005,16, 2213-2220

Packed column

Radiography of the column (transparent PVC)



Pall rings
(H_{Pall} = 800 mm)



4 MellapakPlus 752 elements rotated by 90°
($H_{Mellapak}$ = 800 mm)

D_{bed} = 100 mm

H_{bed} = 1600 mm

Operating conditions

Liquid
- Water
  (μ = 1 mPa.s)
- Glycerine solutions
  (μ = 10 – 20 mPa.s)

Flowrate : u_L = 0 – 25 m³/m².h

Gas = Air

Structured packing

MellapakPlus 752.Y geometrical properties
(Sulzer Chemtech)

Packing element height	0.2 m
Packing element diameter	0.10 m (0.09 m)
Specific surface area	510 m^2/m^3
Void fraction	97.5 %
Corrugation angle	41°
Corrugation base	9.85 mm
Corrugation height	6.50 mm

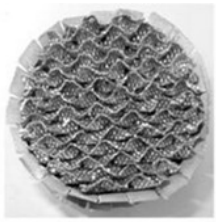
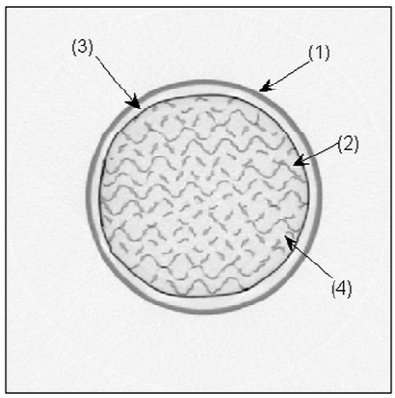
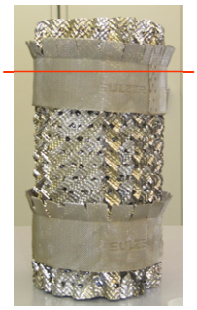


Image of dry packing image



Mellapak 752Y

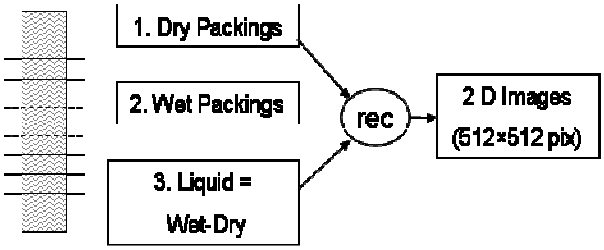


(1) Column wall
(2) Corrugated sheet
(3) Wall wiper
(4) Hole

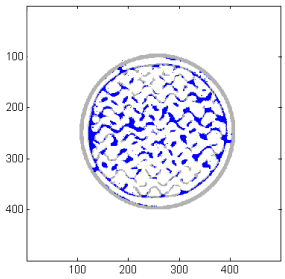
Aferka et al., 2010, Chem. Eng. Sci., 65, 511–516

Images of liquid distribution

Methodology



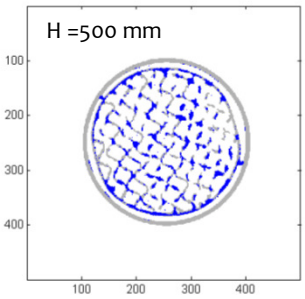
Water
u_L = 23 $m^3m^{-2}h^{-1}$
no gas flow

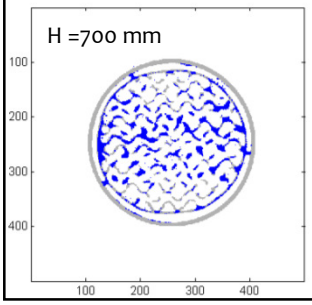


Grey = solid (binary image)
Blue = liquid (binary image)

Aferka et al., 2007, Chem. Eng. Sci., 62, 6076-6080

Liquid distribution in cross sections



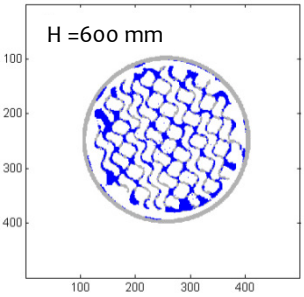


Mellapak 752Y
Water
u_L = 23 $m^3m^{-2}h^{-1}$
No gas flow

Uniform liquid distribution

Film flow predominates

At contact points between sheets, rivulets and/or flooded channels



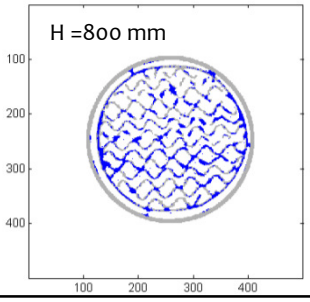
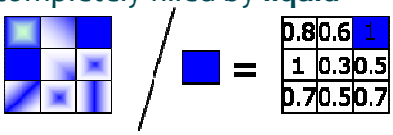


Image processing

Objective = quantitative analysis

1. **Normalisation** (partial volume effect)
   - ⇔ convert grayscale values into **liquid holdup** values
   - → divide each pixel value by the value of a pixel completely filled by **liquid**



→ Distribution of liquid holdup

2. **Counting of interfacial pixels** (liquid – gas)

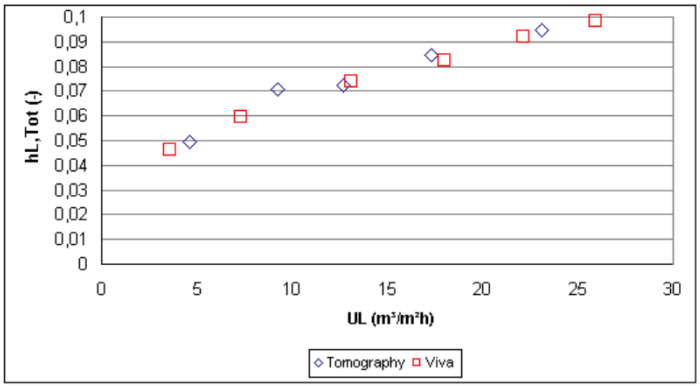
→ Distribution of gas-liquid interfacial area

Viva et al., 2011, Flow Meas Instrum, 22, 279-290

Method validation : liquid holdup

Comparison to experimental data

(global values averaged on 70 sections)



Very different methods:

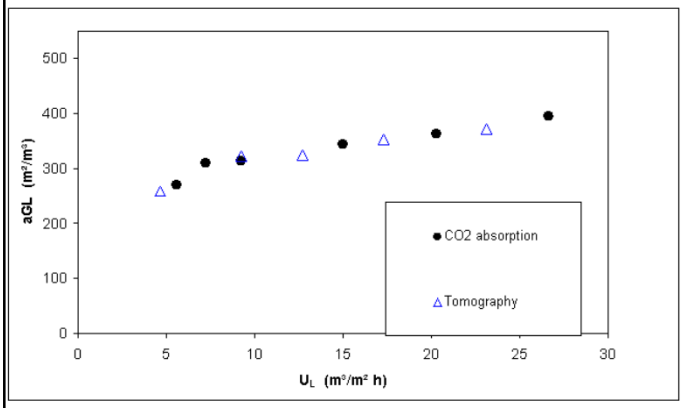
- quick closing valves + weighing
- Tomo + geometrical

Viva, 2008, PhD thesis, University of Pisa

Method validation : G-L interfacial area

Comparison to experimental data

(global values averaged on 70 sections)



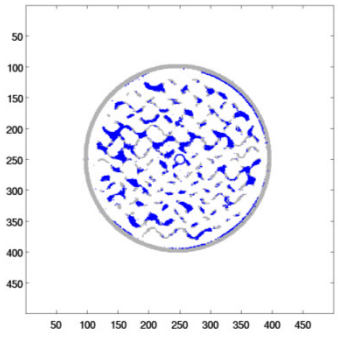
Very different methods:
- chemical
- geometrical

Aferka et al., 2011, Chem. Eng. Sci. , 66, 3413-3422

Influence of liquid viscosity

Mellapak 752Y
H = 300 mm
u_L = 17 $m^3m^{-2}h^{-1}$
u_G = 0

Water
μ = 1 mPa.s

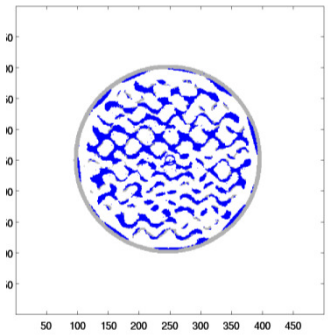


Uniform liquid distribution

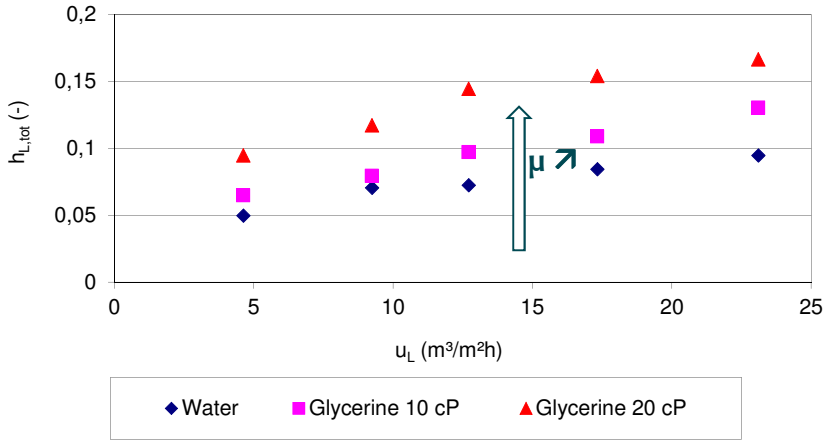
Thicker liquid films

Larger number of flooded channels between sheets

Aqueous solution of glycerine
μ = 10 mPa.s



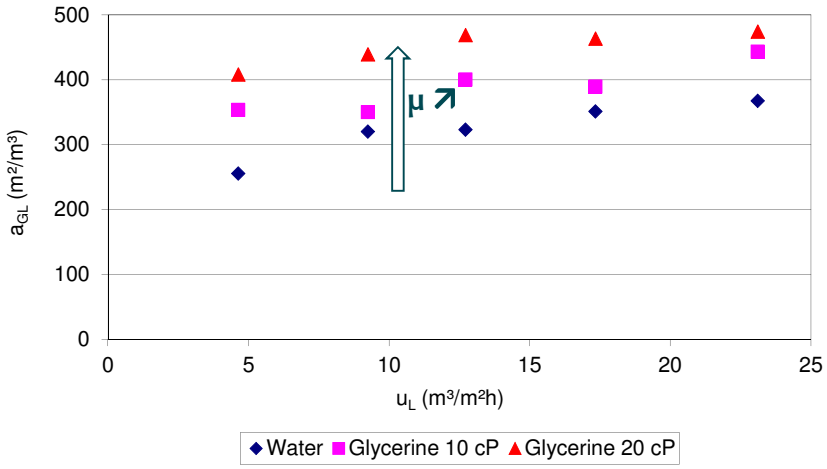
On liquid holdup



Each point = one operating condition
= averaged value over the whole packed bed (70 sections)

Bradtmöller et al., 2015, Ind. Eng. Chem. Res., 52, 2015, 2803-2815

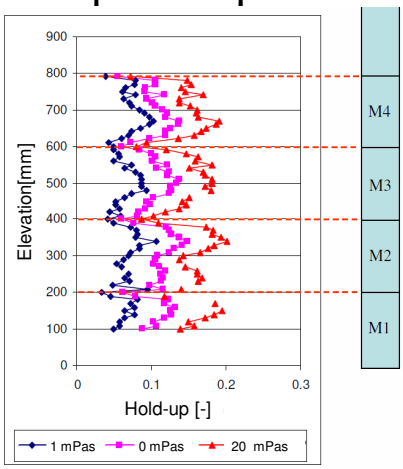
On G-L interfacial area



Each point = one operating condition
= averaged value over the whole packed bed (70 sections)

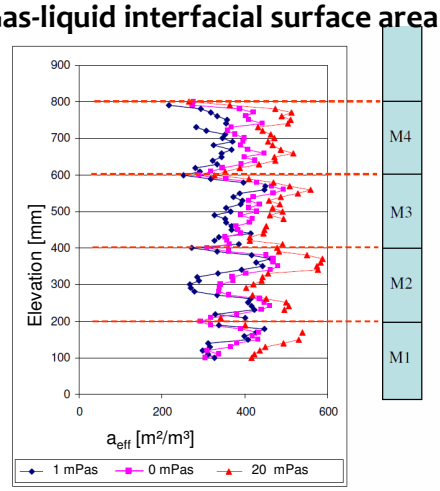
On axial profiles

Liquid hold-up



u_L = 17 m³/m²h

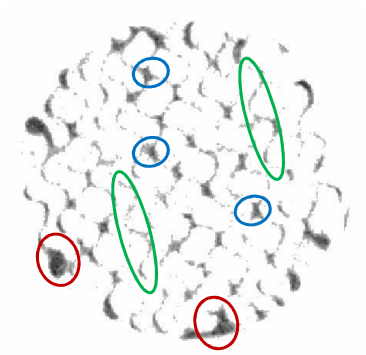
Gas-liquid interfacial surface area



u_L = 17 m³/m²h

70 cross-section images for each operating condition

Liquid flow morphology



Liquid distribution
Binary image

Flow structures

Films
⇔ Ideal morphology

Contact point flow
⇔ Liquid mixing

Flooded channels
⇔ No transfer

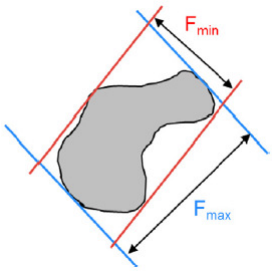
Janzen et al., 2013, Chem. Eng. Sci., 102, 451-460

Flow structure classification

= Iterative method

Step 1 = Separation and labelling of each flow structure

Step 2 = Classification based on the size and shape



SIZE

Minimum and maximum Feret diameters ($\mathbf{F_{min}}$ and $\mathbf{F_{max}}$) computed on all flow structures

$\mathbf{F_{min}}$ and $\mathbf{F_{max}}$ = dimensions of the minimal enclosing parallelogram

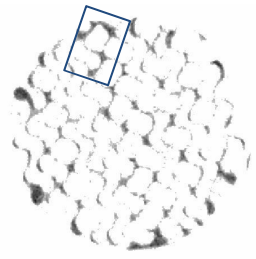
Janzen et al., 2013, Chem. Eng. Sci., 102, 451-460

Flow structure classification

Step 1 = Separation and labelling of flow structures

Limited spatial resolution of the ULG X-ray CT

⇔Distinct flow structures may be adjacent
⇔ « seen » as a single structure



➔ **Criterion for structure splitting**

$$\frac{S_{Flow\_struct}}{S_{Feret\_rect}} < 0.5$$

⇔ Elimination of pixels with the smallest number of neighbors
(erased pixels not lost = arbitrarily added to the main flow pattern = film flow)

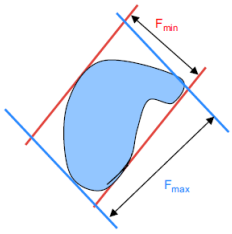
Janzen et al., 2013, Chem. Eng. Sci., 102, 451-460

Flow structure classification

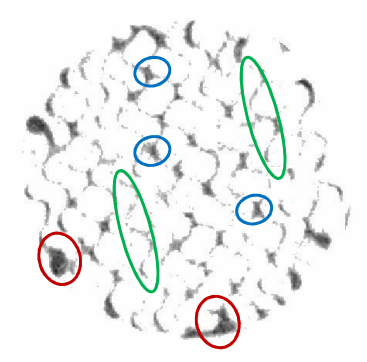
Step 2 = Classification based on the size and shape

Flooded channels

Thickness ≥ distance between packing sheets



$F_{min} > 9$ mm

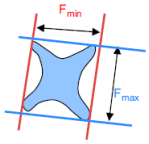


Flow structure classification

Step 2 = Classification based on the size and shape

Contact point flow

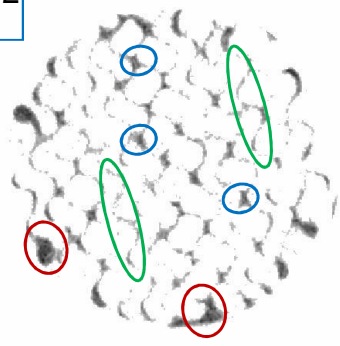
« Round » shape



$\frac{F_{max}}{F_{min}} < 2$

and

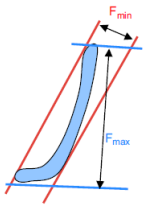
$F_{min} < 9$ mm



Flow structure classification

Step 2 = Classification based on the size and shape

Film flow

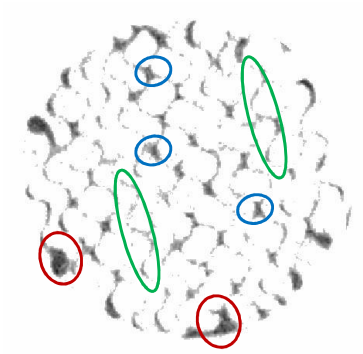


Elongated thin shape

$$\frac{F_{max}}{F_{min}} > 2$$

and

$F_{min} < 9$ mm

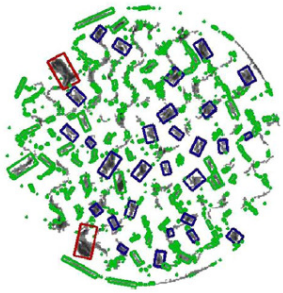


+ all remaining pixels (including erased pixels from structure separation)

Flow morphology: results

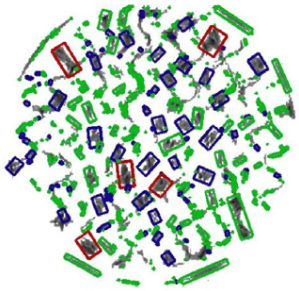
μ = 1 mPa.s
u_L = 17 m³/m²h

Flooded channels



μ = 10mPa.s
u_L = 17 m³/m²h

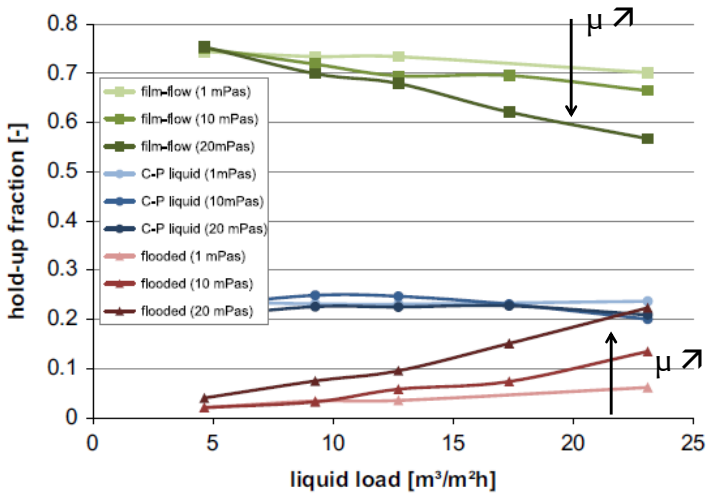
Contact points



μ = 20 mPa/s
u_L = 17 m³/m²h

Films

Flow morphology



Films

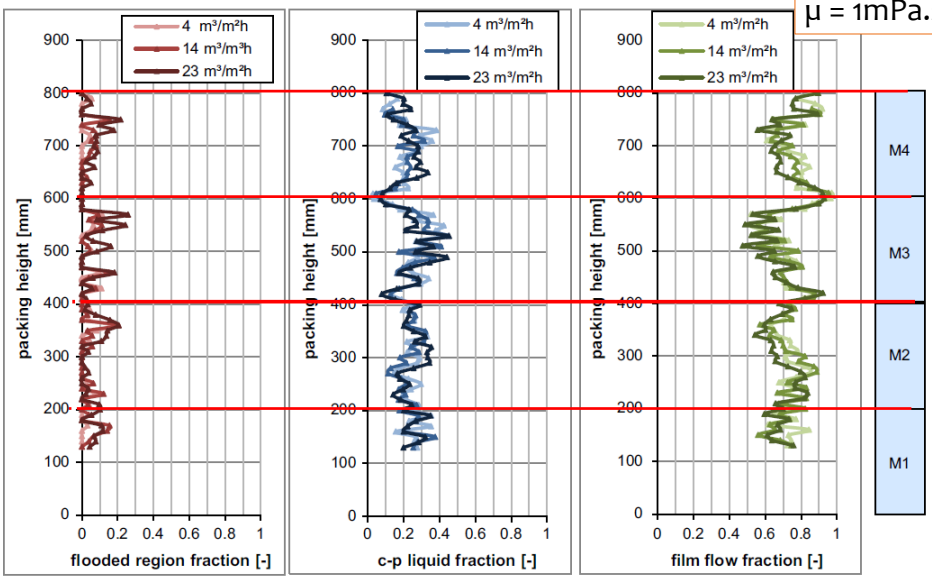
Contact points

Flooded channels

Janzen et al., 2013, Chem. Eng. Sci., 102, 451-460

Influence of liquid flowrate

μ = 1mPa.s



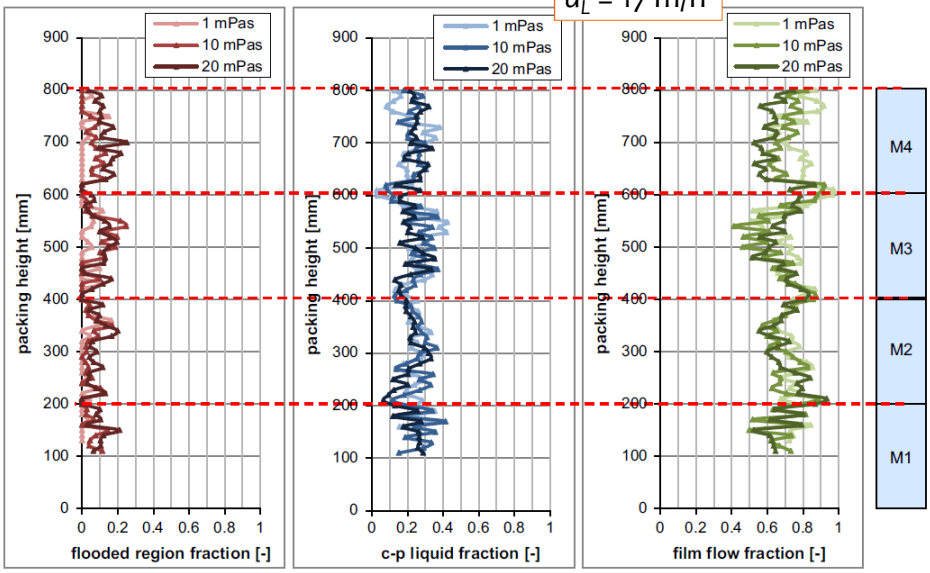
Flooded channels

Contact points

Films

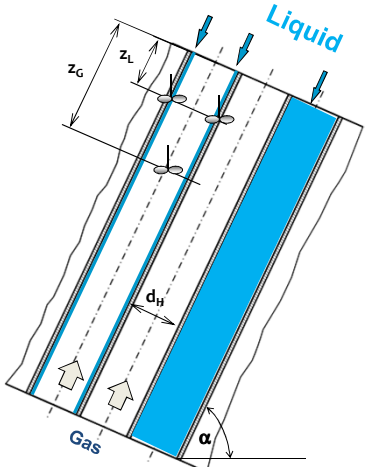
Influence of liquid viscosity

u_L = 17 m/h



Flooded channels **Contact points** **Films**

Hydrodynamic Analogy model



z_G = gas mixing length (packing geometry)

Shilkin et al., 2006, *AIChE J.*, 52, 3055-3066

G-L interfacial surface area ⇔ Wetting efficiency

⇒ fraction of irrigated and non-irrigated channels

Liquid holdup

=> total amount of liquid

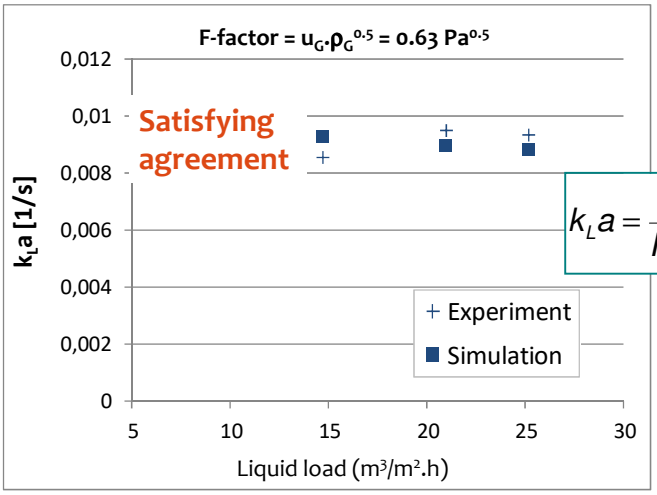
Liquid flow morphology

⇒ Flooded channels fraction

⇒ Contact point flow fraction (⇔ mixing length : z_L)

$$z_L = \frac{\text{dist. between contact points}}{\text{contact point flow fraction}}$$

Model validation : mass transfer



CO_2 desorption from water

$$k_L a = \frac{u_L}{H_{pack}} \ln\left(\frac{x_{top} - x^*_{top}}{x_{bot} - x^*_{bot}}\right)$$

Conclusions

Tomographic measurements

Quantitative assesment of the influence of **liquid load** and **liquid viscosity** on

- G-L interfacial surface area
- Liquid holdup
- Flow morphology (films, rivulets, flooded)

Global values
Spatial distributions

Mass-transfer model

- Based on an Hydrodynamic Analogy
- Validated by mass transfer experiments


Acknowlegments
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